GAU 1621

Practitioner's Docket No. U012693-7 *PATENT*

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Anthony John Oliver, et al Group No.:1621
Serial No.:09/537,250
Filed: March 28, 2000 Examiner:
For: PROCESS FOR DISTILLING FISCHER-TROPSCH DERIVED PARAFFINIC HYDROCARBONS

Assistant Commissioner for Patents
Washington, D.C. 20231

TRANSMITTAL OF CERTIFIED COPY

Attached please find the certified copy of the foreign application from which priority is claimed for this case:

Country: Republic of South Africa

Application Number: PCT/IB99/01448

Filing Date: August 19, 1999

WARNING: "When a document that is required by statute to be certified must be filed, a copy, including a photocopy or facsimile transmission of the certification is not acceptable." 37 C.F.R. 1.4(f) (emphasis added).

CERTIFICATE OF MAILING (37 C.F.R. 1.8a)

I hereby certify that this correspondence is, on the date shown below, being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Date: March 6, 2001

Signature

Janet I. Cord
(type or print name of person certifying)

SIGNATURE OF PRACTITIONER

Reg. No. 33,778

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NOTE: "The claim to priority need be in no special form and may be made by the attorney or agent, if the foreign application is referred to in the oath or declaration, as required by § 1.63." 37 C.F.R. 1.55(a).

Sertifikaat

REPUBLIEK VAN SUID-AFRIKA

Certificate

PATENTKANTOOR

PATENT OFFICE

DEPARTEMENT VAN HANDEL EN NYWERHEID

REPUBLIC OF SOUTH AFRICA

DEPARTMENT OF TRADE AND INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that the documents annexed hereto are true copies of:

Application form P.1, provisional specification and drawings of South African Patent Application No. 98/7599 as originally filed in the Republic of South Africa on 21 August 1998 in the name of SASOL TECHNOLOGY (PROPRIETARY) LIMITED for an invention entitled: "DISTILLATION";

AND it is further certified that Patent Application No. 98/7599 and the invention forming the subject matter of the patent application, together with all priority rights flowing from the patent application under the provisions of the International Convention were duly assigned in accordance with law by SASOL TECHNOLOGY (PROPRIETARY) LIMITED to SCHüMANN-SASOL (SOUTH AFRICA) (PROPRIETARY) LIMITED and SULZER CHEMTECH LIMITED by virtue of Deed of Assignment concluded on 29 July 1999 which Deed of Assignment was duly registered at the Patent Office, Pretoria, on 10 August 1999;

AND it is further certified that inventor Yusuf Omar Dollie was deleted and inventor Mario Roza was added by way of an application for an amendment filed at the South African Patent Office on 11 August 1999 and allowed on 11 August 1999.

Geteken te PRETORIA in die Republiek van Suid-Afrika, hierdie 26th dag van February 2001
Signed at PRETORIA in the Republic of South Africa, this 26th day of February 2001

Registrateur van Patente
Registrar of Patents

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT AND
ACKNOWLEDGEMENT OF RECEIPT
(Section 30(1) Regulation 22)

FORM P.1
(to be lodged in duplicate)

REPUBLIC OF SOUTH AFRICA REVENUE
21.8.98 R 060.00
AKKOMSTE
REPUBLIEK VAN SUID AFRIKA

THE GRANT OF A PATENT IS HEREBY REQUESTED BY THE UNDERMENTIONED APPLICANT ON THE BASIS OF THE PRESENT APPLICATION FILED IN DUPLICATE

21	01	PATENT APPLICATION NO 987599	A&A REF V12946

71	FULL NAME(S) OF APPLICANT(S)

AANSOEKERS VERVANG
APPLICANTS SUBSTITUTED

~~SASOL TECHNOLOGY (PTY) LIMITED~~
1. SCHUMANN - SASOL (SOUTH AFRICA) (PTY) LTD 10/8/99
2. SULZER CHEMTECH LTD

ADDRESS(ES) OF APPLICANT(S)

1 STURDEE AVENUE
ROSEBANK
JOHANNESBURG, REPUBLIC OF SOUTH AFRICA

54	TITLE OF INVENTION

"DISTILLATION"

Only the items marked with an "X" in the blocks below are applicable.

- THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. The earliest priority claimed is

Country: No: Date:

- THE APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO 21 01
- THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON APPLICATION NO 21 01

THIS APPLICATION IS ACCOMPANIED BY:

X	A single copy of a provisional specification of 9 pages
X	Drawings of 1 sheets
	Publication particulars and abstract (Form P.8 in duplicate) (for complete only)
	A copy of Figure of the drawings (if any) for the abstract (for complete only)
X	An assignment of invention
	Certified priority document(s). (State quantity)
	Translation of the priority document(s)
	An assignment of priority rights
	A copy of Form P.2 and the specification of RSA Patent Application No 21 01
X	Form P.2 in duplicate
X	A declaration and power of attorney on Form P.3
	Request for ante-dating on Form P.4
	Request for classification on Form P.9
	Request for delay of acceptance on Form P.4
	Extra copy of informal drawings (for complete only)

74	ADDRESS FOR SERVICE: Adams & Adams, Pretoria

Dated this **21** day of **August 1998**

ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS

The duplicate will be returned to the applicant's address for service as proof of lodging but is not valid unless endorsed with official stamp

REGISTRAR OF PATENTS, DESIGNS, TRADE MARKS AND COPYRIGHT
1998 -08- 21
REGISTRATEUR VAN PATENTE, MODELLE, HANDELSMERKE EN OUTEURSREG
REGISTRAR OF PATENTS

A&A P201

ADAMS & ADAMS
PRETORIA

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

FORM P.3

DECLARATION AND POWER OF ATTORNEY

(Section 30 - Regulation 8, 22(i)(c) and 33)

PATENT APPLICATION NO		
21	01	98/7599

A&A Ref: V12946 GSK

LODGING DATE	
22	21 AUGUST 1998

FULL NAME(S) OF APPLICANT(S)	
71	SASOL TECHNOLOGY (PROPRIETARY) LIMITED

FULL NAME(S) OF INVENTOR(S)		
72	1. OLIVIER, Anthony John	2. RICHTER, Ferdinand
	3. DUCKITT, Charles	4. RAMDUTH, Ashwin
	5. ADAMS, Vernon Jeremay	6. MOODLEY, Vinothen
	7. CALDER, Roy Alexander	8. ROZA, Mario

EARLIEST PRIORITY CLAIMED	COUNTRY		NUMBER		DATE	
	33	--	31	--	32	--

NOTE: The country must be indicated by its International Abbreviation - see schedule 4 of the Regulations

TITLE OF INVENTION	
54	"DISTILLATION"

REGISTRAR OF PATENTS, DESIGNS, TRADE MARKS AND COPYRIGHT
1999 -11- 22
REGISTRATEUR VAN PATENTE, MODELLE, HANDELSMERKE EN OUTEURSREG

* I/We VAN DER MERWE, Martha Magdalena

hereby declare that :-

~~1. I/we am/are the applicant(s) mentioned above;~~

** 2. I/we have been authorized by the applicant(s) to make this declaration and have knowledge of the facts herein stated in the capacity of Authorized Signatory of the applicant(s);

*** 3. the inventor(s) of the abovementioned invention is/are the person(s) named above and the applicant(s) has/have acquired the right to apply by virtue of an assignment from the inventor(s);

4. to the best of my/our knowledge and belief, if a patent is granted on the application, there will be no lawful ground for the revocation of the patent;

**** ~~5. this is a convention application and the earliest application from which priority is claimed as set out above is the first application in a convention country in respect of the invention claimed in any of the claims;~~ and

6. the partners and qualified staff of the firm of **ADAMS & ADAMS**, patent attorneys, are authorised, jointly and severally, with powers of substitution and revocation, to represent the applicant(s) in this application and to be the address for service of the applicant(s) while the application is pending and after a patent has been granted on the application.

SIGNED THIS 7th DAY OF September 1999

Company Name: SASOL TECHNOLOGY (PROPRIETARY) LIMITED
Full Names: VAN DER MERWE, Martha Magdalena
Capacity: Authorized Signatory

(no legalization necessary)

* In the case of application in the name of a company, partnership or firm, give full names of signatory/signatories, delete paragraph 1, and enter capacity of each signatory in paragraph 2.
** If the applicant is a natural person, delete paragraph 2.
*** If the right to apply is not by virtue of an assignment from the inventor(s), delete "an assignment from the inventor(s)" and give details of acquisition of right.
**** For non-convention applications, delete paragraph 5.

A & A Ref No: V12946

ADAMS & ADAMS
PATENT ATTORNEYS
PRETORIA

FORM P6

REPUBLIC OF SOUTH AFRICA
Patents Act, 1978

PROVISIONAL SPECIFICATION

(Section 30 (1) - Regulation 27)

21	01	OFFICIAL APPLICATION NO	22	LODGING DATE
		98/7599		21 AUGUST 1998

71	FULL NAME(S) OF APPLICANT(S) *

~~SASOL TECHNOLOGY (PROPRIETARY) LIMITED~~

AANSOEKERS VERVANG
APPLICANTS SUBSTITUTED
10/8/99

1. SCHUMANN-SASOL (SOUTH AFRICA) (PTY) LTD
2. SULZER CHEMTECH LTD

72	FULL NAME(S) OF INVENTOR(S)

1. ANTHONY JOHN OLIVIER
2. FERDINAND RICHTER
3. CHARLES DUCKITT
4. ASHWIN RAMDUTH
5. VERNON JEREMAY ADAMS
6. VINOTHEN MOODLEY
7. ROY ALEXANDER CALDER
8. MARIO ROZA

54	TITLE OF INVENTION

"DISTILLATION"

THIS INVENTION relates to distillation. More particularly, the invention relates to a process for distilling paraffinic hydrocarbons, particularly Fischer-Tropsch derived paraffinic hydrocarbons.

According to the invention, there is provided a process for distilling paraffinic hydrocarbons, which process comprises

feeding a Fischer-Tropsch derived paraffinic hydrocarbon feedstock comprising heavy paraffinic hydrocarbons and, optionally, light and/or medium paraffinic hydrocarbons, into a distillation column;

operating the distillation column to produce usable wax products; and

withdrawing from the distillation column an overhead stream, a bottom stream, and at least one side stream, with the bottom stream and/or the side stream comprising usable wax products.

By 'usable' in respect of the wax products is meant that the wax products are non-thermally degraded. These products will normally also meet stringent specifications with respect to properties such as congealing point, methyl-ethyl-ketone solubles, penetration, differential scanning calorimetry curves etc.

By 'Fischer-Tropsch derived' is meant paraffinic products obtained by subjecting a synthetic gas comprising carbon monoxide (CO) and hydrogen (H_2) to Fischer-Tropsch reaction conditions in the presence of an iron-based, a cobalt-based or an iron/cobalt-based Fischer-Tropsch catalyst. The products of the Fischer-Tropsch reaction are predominantly n-paraffinic, although some isomers, olefins, oxygenates and other functional groups may also be produced. The products from the Fischer-Tropsch reaction have a wide boiling range. Prior to using the products from the Fischer-Tropsch reaction as a feedstock for the present process,

they may optionally be hydrogenated. Such hydrogenation may be effected by contacting the Fischer-Tropsch reaction products with hydrogen in the presence of a hydrogenation catalyst, at elevated temperature and pressure, in known fashion.

The Fischer-Tropsch reaction conditions include using a relatively low reaction temperature in the range 180-300°C, typically 210-260°C, so that a so-called low temperature Fischer-Tropsch synthesis is employed, and the Fischer-Tropsch reaction is typically effected in a fixed or slurry bed reactor.

The feedstock may comprise, in addition to the heavy paraffinic hydrocarbons, the light and the medium paraffinic hydrocarbons. The feedstock could thus typically have a true boiling point curve as indicated in Table 1:

TABLE 1: True boiling point (TBP) curve of a typical Fischer-Tropsch derived feedstock

Mass %	TBP (°C)
1	142
5	169
10	195
30	313
50	417
70	550
90	716
95	757
98	831

The feedstock typically comprises hydrocarbon molecules in the range C_{3+} to C_{220+}. Products with carbon ranges of C_{20-}, C_{10} to C_{40} and C_{15} to C_{220} or higher, are deemed light, medium and heavy hydrocarbons respectively.

The distillation column can be operated to produce paraffins (C_{23-}), medium wax (C_{20} to C_{38}), and hard wax (C_{30+}) or combinations thereof, with these products then being withdrawn in the overhead stream, the bottom stream or in the at least one side stream. All the wax products produced will thus be usable wax products as hereinbefore defined.

Preferably, however, a plurality of side streams are withdrawn from the column, with each side stream comprising a component of the paraffins, a component of the medium wax, or a component of the hard wax, or combinations thereof.

The distillation column is preferably operated under vacuum. Thus, the pressure in the column may be in the range of 1 to 12 mbar(a), typically 8 to 10 mbar(a). The temperature in the column sump may then be in the range of 190°C to 340°C, typically in the range of 295°C to 300°C.

It is preferred to operate the column under vacuum, to prevent, or at least inhibit, thermal degradation of the feedstock and the products. Such thermal degradation will have a negative effect on the properties of the products produced, such as on the congealing point, penetration and oil content of the wax products.

The process may include feeding stripping steam into the distillation column, to adjust the relative volatility of components in the feedstock. The process may also include feeding one or more of the side streams through a stripping stage. It is envisaged that steam stripping can be used to adjust the front end volatility of the products, thereby to aid in product quality.

The distillation column may comprise structured packing typically having a surface area (in m^2) to volume (in m^3) ratio of 125:1 to 750:1, e.g. 250:1, 350:1 or 500:1, or any other intermediate value.

This packing and internal arrangement of the distillation column produces a very low pressure drop and minimal entrainment, while ensuring that the required separation is achieved. Typically, five theoretical stages are provided, with the respective stages each containing the packing and the internal arrangement and each stage being located between draw points for the overhead, side and bottom streams from the column. The packings of the various stages can have the same surface area to volume ratios, or the

surface area to volume ratios of the packings of at least some of the stages can be different. The internal arrangement minimizes the residence time within the distillation column, thus reducing the amount of thermal cracking of the products produced.

The process of the invention thus employs multiple side streams with separation stages in the column between the withdrawal of the side streams, to split wax fractions.

With the process of the invention, the Fischer-Tropsch derived feedstock is thus fractionated into product streams having unique properties. One of these properties is the congealing point, which can thus be used to control the operation of the distillation column. However, instead, or additionally, other unique properties, such as methyl-ethyl-ketone (MEK) / methyl-isobutyl-ketone (MIBK) solubles, penetration at 25°C, carbon distributions, etc. can be used to control distillation operation. The number of side streams from the column are determined by the properties of the products and by product purity desired. There is, in principle, no restriction on the maximum number of side stream product draws other than the fact that the accumulated pressure drop of the internals must be limited.

The invention will now be described by way of example, with reference to the accompanying drawing and non-limiting example.

In the drawing, reference numeral 10 generally indicates, in simplified flow diagram form, a process according to the invention for distilling paraffinic hydrocarbons.

In the drawing, reference numeral 10 generally indicates a process according to the invention, for distilling a Fischer-Tropsch derived light, medium and heavy paraffinic hydrocarbon feedstock.

The process 10 includes a distillation column 12 having six vertically staggered packing stages 14, 16, 18, 20, 22 and 24. Each packing stage comprises high performance structured packing

and associated internals such as structured packing having a surface area (in m^2) to volume (in m^3) ratio of 125:1, 250:1, 350:1, 500:1 or 750:1, or any appropriate intermediate value..

A feed line 26 leads into the bottom of the distillation column 12, as does a stripping steam feed line 28. Into the line 26 leads a light (C_{20-}) hydrocarbon line 30, a medium (C_{10} - C_{40}) hydrocarbon line 32 and a heavy (C_{15} - C_{220+}) hydrocarbon line 34.

The feed line 26 and the stripping steam feed line 28 lead into the column below the lowermost packing stage 14.

A bottoms line 36 leads from the bottom of the column 12.

A side stream line 38 leads from the column between the packing stages 14, 16 to a stripping column 40, with a stripping steam line 42 leading into the bottom of the column 40. The column 40 comprises a packing stage 44 comprising sieve trays. A product line 46 leads from the bottom of the column 40, while a return line 48 leads from the top of the column 40. The return line 48 returns to the column 12 between the packing stages 16, 18.

A side stream withdrawal line 50 leads from the distillation column between the packing stages 16, 18 into a stripping column 52 having a packing stage 54 comprising sieve trays. A product withdrawal line 56 lead from the bottom of the column 52, while a return line 58 leads from the top of the column 52 back to the distillation column 12 between the packing stages 18, 20.

A side stream withdrawal line 60 leads from the column 12 between the packing stages 18, 20. The line 60 leads into the top of a stripping column 62 having a packing stage 64 comprising sieve trays. A product withdrawal line 66 leads from the bottom of the column 62, while a return line 68 leads from the top of the column 62 back to the distillation column 12 between the packing stages 20, 22.

A side stream withdrawal line 70 leads from the distillation column 12 between the packing stages 20, 22. The line 70 leads

into a stripping column 72 having a packing stage 74 comprising sieve trays. A product withdrawal line 76 leads from the bottom of the column 72, while a return line 78 leads from the top of the column 72 back to the distillation column 12, between the packing stages 22, 24.

A side stream/product withdrawal line 80 leads from the distillation column 12 between the packing stages 22, 24, and is fitted with a recycle line 82 returning to the distillation column 12 above the packing stage 24.

An overheads line 84 leads from the top of the column.

In use, a Fischer-Tropsch derived light, medium and heavy hydrocarbon feedstock is fed, along the flow line 26, into the bottom of the distillation column 12. The distillation column 12 is typically operated at a pressure of 8-10 mbar(a) and at a temperature, measured in the column sump, of about 295-300°C.

Usable wax products, such as medium wax (C_{20} - C_{38}) and hard wax (C_{30+}) are produced in the column 12.

The products withdrawn along the lines 36, 46, 56, 66, 76, 80 and 84 typically comprise C_{35+}, C_{25} - C_{40}, C_{20} - C_{30}, C_{19} - C_{23}, C_{18} - C_{20}, C_{17-} and C_{5-} respectively.

Stripping steam lines 86 lead into the bottoms of each of these stripping columns 52, 62, 72.

The following non-limiting example was also conducted, in a simulation of the process 10:

EXAMPLE

The feedstock entering the column 12 along the line 26 comprised light hydrocarbons (also known and referred to as Cold Condensate (CC)), medium hydrocarbons (also known and referred to as Hot Condensate (HC)) and heavy hydrocarbons (also known and referred to as Reactor Waxes (RW)). All the hydrocarbons were Fischer-

Tropsch derived. Thus, each component of the feedstock was a blend of the respective products from both fixed and slurry bed reactor Fischer-Tropsch processes. The blend ratio in this example was:

CC = 28.8 % by mass
HC = 17.2 %
RW = 54.0 %

The above blend ratio was dictated by the production rates from the existing fixed and slurry bed Fischer-Tropsch reactor processes. Blend ratios of only slurry bed or fixed bed products can be used as well.

The number of side streams from the column 12 are determined by the properties of the product or the by-product purity desired.

There is no restriction on the maximum number of side product streams other than the fact that the accumulated pressure drop of the internals must be limited. If unlimited, energy loss and thermal cracking can be so significant that the process becomes technologically and/or economically non-viable.

Table 2 hereunder shows the streams produced, the desired congealing point (CP) range and typical CP values obtained.

	Product	Name	CP Desired Range (°C)	Typical CP obtained (°C)
Overhead Stream 84	C_{5-}	Gas	n/a	n/a
Stream 80	C_{17-}	C_{17}-Paraffins	n/a	n/a
Stream 76	C_{18}-C_{20}	C_{18}-C_{20} Paraffins	25-30	28
Stream 66	C_{19}-C_{23}	Waksol	35-40	38
Stream 56	C_{20}-C_{30}	Medium Wax 1	50-55	53
Stream 46	C_{25}-C_{40}	Medium Wax 2	60-65	64
Bottom Stream 36	C_{35+}	Hard Wax	65+	98

The yield of the above streams on a mass basis as a percentage of the feed was approximately:

Overhead Stream 84	=	1.0 %
Stream 80	=	27.6 %
Stream 76	=	5.8 %
Stream 66	=	4.5 %
Stream 56	=	6.9 %
Stream 46	=	11.4 %
Bottom Stream 36	=	42.8 %

The column 12 was operated at 9 mbar(a) using a four stage steam ejector for its vacuum system. It had six packed beds with low pressure drop structured packing, each bed comprising of Sulzer 250Y (trade mark) packing available from Sulzer Chemtech Ltd, PO Box 65, CH-8404, Winterthur, Switzerland. Some side streams had side stripper columns as indicated in the drawing. Low pressure (2.4 bar_g) steam was injected into both the bottom of the main fractionator and the side stripper columns to aid in separation.

The process 10 permits a light, medium and heavy Fischer-Tropsch derived feedstock to be distilled into normal usable product ranges using a single column with multiple product side streams. This has hitherto not been possible due to high pressure drops associated with conventional pattern of distillation columns. The wax products produced are usable wax products.

The process 10 is capable of producing a wide range of narrow cuts, and also has substantial flexibility.

DATED THIS 21ST DAY OF AUGUST 1998

ADAMS & ADAMS
APPLICANT'S PATENT ATTORNEY